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## Research Article

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# Carbonation-Based Recycling of Concrete Wash Water for Pollution Control and CO<sub>2</sub> Sequestration

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## ABSTRACT

This study proposes an integrated cleaner-production route that combines accelerated CO<sub>2</sub> mineralization with wastewater recycling for the treatment of concrete wash water (CWW) from ready-mixed concrete production. CWW was carbonated through gas-liquid mixing to produce carbonated concrete wash water (CCWW), which was subsequently reused as mixing water for cement mortar. The results showed that, compared with the potable-water reference group, the use of CCWW reduced the drying shrinkage by 52.9%, increased the compressive strength by 13.0%, and decreased the flowability by 12.6%. In addition, quantitative substitution tests indicated that when the replacement ratio of mixing water by CCWW was lower than 50%, the relevant performance requirements could still be satisfied. Furthermore, optimization of the solid/liquid ratio, CO<sub>2</sub> flow rate, and cooling-water temperature by response surface methodology increased the CO<sub>2</sub> absorption rate by 14.1%, reaching 18.6 g/min. These findings demonstrate that accelerated carbonation of CWW provides a feasible route for simultaneous wastewater reutilization and CO<sub>2</sub> sequestration.

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**Keywords:** Concrete wash water; Carbon sequestration; Wastewater recycling; Optimization.

## **1. Introduction**

Concrete wash water (CWW) refers to the wastewater generated during concrete production. This primarily arises from the processes of concrete mixing, transportation and cleaning of equipment after the pouring process. The production of one cubic meter of concrete generates approximately 350 liters of CWW, resulting in the annual production of hundreds of millions tons of CWW(Monkman et al., 2021). The CWW typically contains a substantial quantity of hydrated cement particles and aggregates, resulting in a pH value exceeding 12. The discharge of untreated CWW into aquatic ecosystems can significantly alter the chemical composition of the receiving water and cause environmental pollution. Meanwhile, ready-mix concrete plants also attempt to recycle CWW in concrete production. However, it is often necessary to dilute the CWW with potable water to ensure that the concrete properties comply with the required standards(Ghraid et al., 2020). This is due to the complex composition, high solid content, and non-uniform distribution of physical phases in the CWW. Concrete produced with CWW tends to have a shorter setting time, lower flowability, and adverse effects on the development of pore structure, as well as a reduction in strength(Meena and Luhar, 2019,Peighambarzadeh et al., 2020,Vaičiukynienė et al., 2021). Therefore, the use of CWW and diluted CWW is prohibited in the production of high-performance concrete(Ahmed et al., 2021,Al-Jabri et al., 2011,Asadollahfardi et al., 2016,Fang et al., 2020,Woyciechowski et al., 2021). The conventional treatment of CWW is shown in

Fig. 1. The effective recycling of CWW represents a significant challenge for the construction industry.

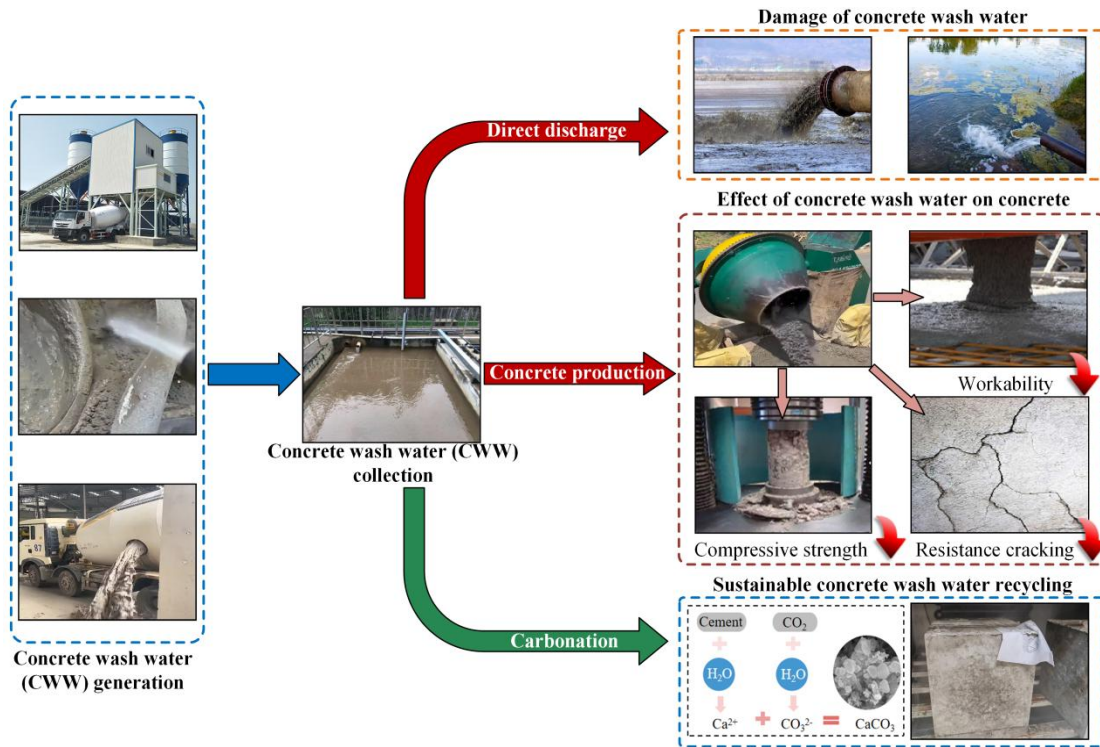


Fig. 1 Three treatments for CWW

In the context of global warming, carbon sequestration technology has gradually emerged as a pivotal strategy for mitigating climate change (Bukar and Asif, 2024, Ho et al., 2025, Zhang et al., 2022). Concrete, as the most widely used building material in the world, contributes significantly to CO<sub>2</sub> emissions during its production and use (Chen et al., 2024, Wang and Fan, 2025). However, concrete also holds great potential for carbon sequestration (Liu et al., 2024, Ridha et al., 2022). The data indicate that approximately 1.4 gigatons of carbon were sequestered between 1930 and 2013, offsetting more than 40% of the CO<sub>2</sub> emissions from cement production during the same period (Xi et al., 2016). Consequently, carbonation has emerged as a pivotal area of investigation within the concrete domain, intending to leverage the chemical reaction between CO<sub>2</sub> and cement constituents to augment the physical and chemical attributes

of concrete(Cheng et al., 2025,Fang et al., 2023). Recent studies have shown that carbonation treatment can effectively improve the physical and mechanical properties of recycled aggregates and the resulting recycled concrete by refining pore structure and enhancing interfacial transition zone (ITZ) bonding(Yajing et al., 2025). Abid et al.(Abid et al., 2015) conducted a study which revealed that concrete subjected to CO<sub>2</sub>-enriched conditions exhibited significant improvements in terms of durability and compressive strength. With the increasing focus on sustainable development, the recycling of CWW has gained attention. Sean et al.(Monkman et al., 2022) carried out research which showed that the CWW can improve the properties of cement mortar when used to replace potable water. Recent studies have extended this topic to data-driven prediction. For example, Farouk et al. showed that machine learning could effectively predict the compressive strength of concrete made with treated wastewater, highlighting the importance of water-quality variability(Farouk et al., 2025). In addition, Mehdizadeh et al. explored the feasibility of using concrete washout water as the liquid medium for wet carbonation, highlighting its potential for simultaneous wastewater reutilization and CO<sub>2</sub> sequestration(Mehdizadeh et al., 2025). David et al.(Suescum-Morales et al., 2022) investigated the treatment of CWW and its application in cement production, showing that the components in the wastewater can be effectively transformed through the carbonation process, reducing environmental pollution. In this process, the composition and characteristics of CWW directly influence the carbonation effect and the final properties of concrete. Thus, the study of the carbonation characteristics of CWW and its effect on concrete properties is of great theoretical and

practical value.

The utilization of carbon dioxide in the treatment of CWW presents an opportunity for carbon removal, which can ameliorate the environmental impact of concrete production (Hu et al., 2023). However, previous studies have mainly focused either on the direct reuse of concrete wash water or on carbonation treatment alone, and the integration of carbonation treatment, compositional evolution analysis, reuse performance evaluation, and process optimization has rarely been reported. Therefore, this study proposes an accelerated carbonation-based recycling route for CWW. The main objectives are to achieve wastewater treatment and recycling of CWW through accelerated carbonation, while simultaneously enhancing CO<sub>2</sub> uptake and sequestration during the treatment process. In addition, the reuse performance of carbonated concrete wash water (CCWW) in cement mortar was evaluated to preliminarily assess its resource utilization potential. Specifically, the effects of carbonation on the composition of CWW and the influence of CCWW on cement mortar properties were first analyzed. Then, the effects of solid/liquid ratio, CO<sub>2</sub> gas flow rate, and coolant temperature on the CO<sub>2</sub> absorption rate of CWW were investigated, and optimization analysis was performed to determine the optimum operating conditions. The novelty of this work lies in establishing an integrated framework that links carbonation treatment, CCWW characterization, mortar reuse performance, and operating-parameter optimization. It should be noted that the RSM part of this study mainly focuses on the optimization of carbonation operating parameters to improve CO<sub>2</sub> uptake efficiency and establish a controllable preparation process for CCWW, rather than directly optimizing

mortar performance.

## 2. Materials and methods

### 2.1. Testing material

To reduce the variability associated with field-derived wash water, simulated concrete wash water (CWW) was prepared in the laboratory. Specifically, 0.15 kg of ordinary silicate cement was added to 1.0 kg of potable water (solid/liquid ratio = 150:1000, by mass) and thoroughly stirred. The slurry was then allowed to hydrate for 3 h at room temperature and used immediately for carbonation tests. The composition and properties of the actual CWW vary significantly due to the fact that it may be affected by a number of factors, such as source, treatment process, and storage time. Therefore, the use of simulated CWW in this study was intended to establish a simplified, controllable, and reproducible experimental system for investigating the carbonation mechanism and the preliminary feasibility of the proposed treatment-and-reuse route. However, this simplification limits the direct representativeness of the present results for real industrial wash water. Accordingly, the current work should be regarded as a preliminary exploration under laboratory conditions, and further validation using real CWW collected from concrete plants remains an essential step for future research. The test sand conformed to the Chinese ISO standard, with a fineness modulus of 3.0. The CO<sub>2</sub> gas used had a purity of  $\geq 99.5\%$ . The cement was ordinary silicate cement (P·I 42.5), and the chemical analysis results of the benchmark cement clinker and its mineral composition (%) are shown in Table 1.

**Table 1** Chemical analysis results and mineral composition of ordinary silicate cement

| Mineral | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | CaO | MgO | SO <sub>3</sub> |
|---------|------------------|--------------------------------|--------------------------------|-----|-----|-----------------|
|---------|------------------|--------------------------------|--------------------------------|-----|-----|-----------------|

| composition         |                     |       |                  |                  |                  |                   |
|---------------------|---------------------|-------|------------------|------------------|------------------|-------------------|
| Proportion (%)      | 20.02               | 4.97  | 3.81             | 63.17            | 3.87             | 0.40              |
| Mineral composition | Na <sub>2</sub> Oeq | f-CaO | C <sub>3</sub> S | C <sub>2</sub> S | C <sub>3</sub> A | C <sub>4</sub> AF |
| Proportion (%)      | 0.60                | 0.89  | 58.94            | 15.31            | 6.71             | 11.58             |

## 2.2. Test equipment and carbonation method

A lab-scale carbonation recovery unit was built for the accelerated carbonation of CWW (Fig. 2). The system comprises (I) a gas circulation systems (CO<sub>2</sub> storage tank, CO<sub>2</sub> pressure-reducing valve, CO<sub>2</sub> flow meter, air pump and gas piping), (II) a liquid circulation systems (submersible pump, fluid circulation catheter and conduit outlet), and (III) a control systems (air pressure sensor, flow sensor temperature sensor pH sensor and controllers), (IV) other (wash water, cooling circuit, carbon sequestration tank). During operation, the submersible pump continuously recirculates CWW within the tank. CO<sub>2</sub> is injected at the pump outlet, where it is dispersed into fine bubbles and mixed with the circulating CWW, thereby accelerating CO<sub>2</sub> dissolution and the carbonation reactions (Eqs. (1)-(4)). To mitigate the heat release during carbonation, a cooling loop (cooling circuit and circulation tank in Fig. 2) was employed; the coolant temperature was set according to the experimental design and monitored by a temperature sensor immersed in the CWW. In this study, the coolant temperature factor refers to the operating setpoint of the laboratory cooling loop used to regulate the exothermic carbonation process, rather than the direct actual temperature of the reacting CWW at all times.

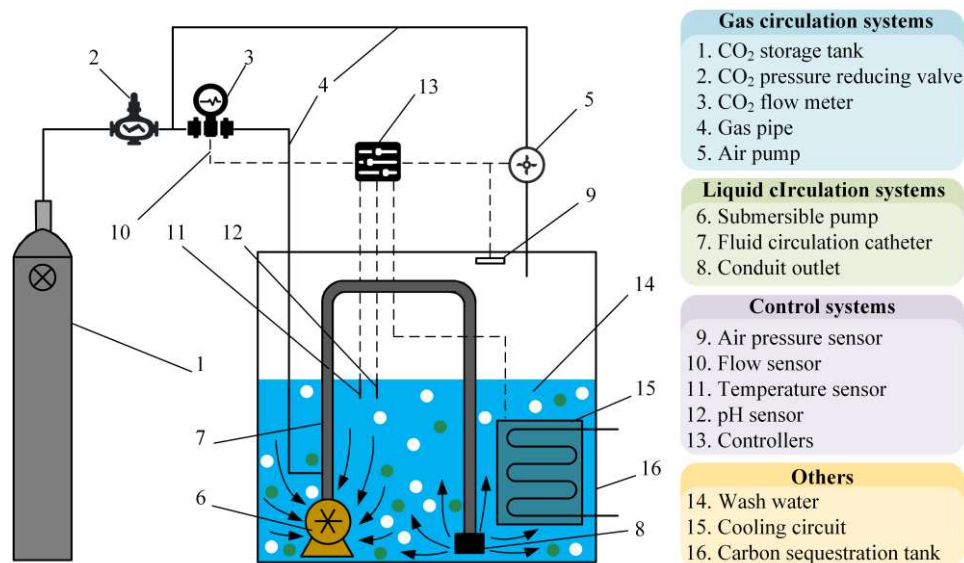
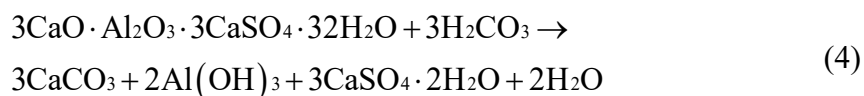
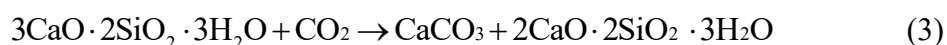
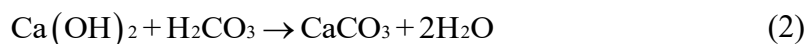


Fig. 2 CWW carbonation recovery unit

When the pressure reaches a pre-set limit, the pressure sensor sends an electrical signal to the controller. The controller then activates the pump to return the CO<sub>2</sub> gas to the tank just before the flowmeter. The flowmeter controls the CO<sub>2</sub> gas flow rate and returns the gas to the tank to continue the reaction.



Each carbonation run followed the same procedure: (a) a freshly prepared CWW batch was poured into the carbonation tank; the pH meter electrode, thermometer and conductivity probe were inserted, and the sealed tank was placed on an electronic balance (accuracy 0.1 g); (b) the submersible pump and cooling circulation were started and the balance was tared; (c) CO<sub>2</sub> was introduced at the prescribed flow rate via the flow meter to initiate carbonation; (d) The reaction was terminated when the pH reached

approximately 7 and remained stable. This operational endpoint was further supported by the subsequent XRD and TG analyses, which confirmed substantial consumption of  $\text{Ca(OH)}_2$  and significant formation of  $\text{CaCO}_3$  after carbonation.

### *2.3. Test process and test methods*

The CWW preparation, carbonation and testing procedure of the CWW is shown in Fig. 3. The CWW was first prepared with a solid/liquid ratio of 150:1000, and then hydrated for three hours with appropriate stirring. After hydration, the mixture was poured into the carbonation tank for carbonation. The CWW before and after carbonation was divided into two parts. One portion was subjected to solid/liquid separation using a vacuum filter. The solid residue was used for solid phase analysis. The filtered liquid was used for liquid phase analysis. The second part of the CWW was used to test the workability of the cement grits. Finally, Response Surface Methodology (RSM) was performed by varying the solid/liquid ratio,  $\text{CO}_2$  gas flow rate, and coolant temperature. The total carbon fixation reaction time was determined by stabilizing the pH using a pH meter and the increase in mass was measured on an electronic scale to calculate the fixation rate. Three parallel tests were conducted for each set of conditions, and the average value was used as the test result.

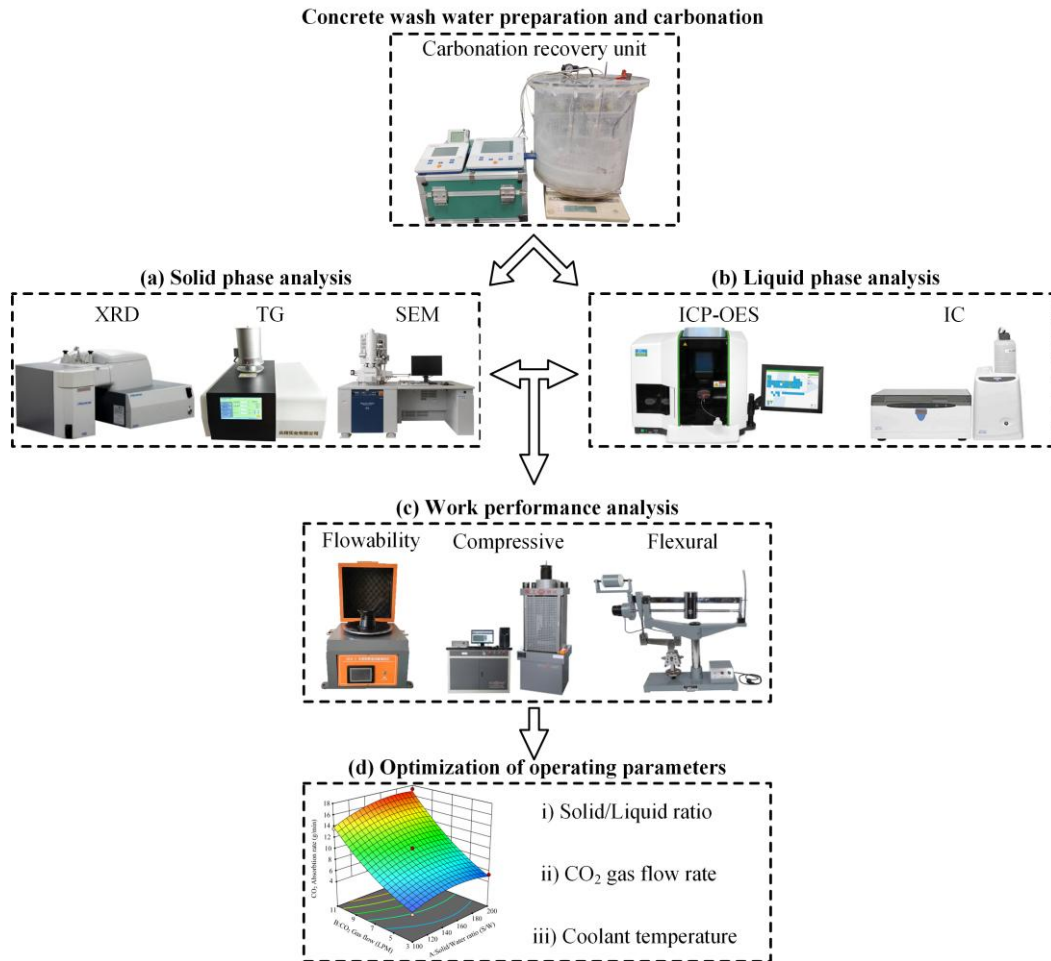


**Fig. 3** CWW sample preparation and test procedures

### 2.3.1. Solid phase analysis test

The dried solid components of the CWW were subjected to solid phase analysis, including XRF, XRD, SEM-EDS, TG analysis and particle size analysis. Samples were separately vacuum filtered using a 0.2  $\mu\text{m}$  organic filter membrane. The filter residue was then placed in a vacuum drying oven and dried at a constant temperature of 50°C until its mass remained constant. The crystalline phase composition was analyzed using an Ultima IV X-ray diffractometer, and the surface morphology of the samples was observed with a Zeiss Sigma 300 scanning electron microscope. The composition of the surface morphology of the samples was further analyzed using an XFlash 6I30 spectrometer. The TG-DTG test was conducted in a nitrogen atmosphere at a flow rate

of 30 mL/min, with a temperature range of 50°C to 900°C and a heating rate of 20°C/min.

### 2.3.2. *Liquid phase analysis test*

ICP-OES and IC ion chromatography were used to analyze the concentration of major ions in the CWW before and after carbonation. To assess the changes in ionic composition, the analysis was conducted based on the JCT 2647-2021 "Specification for waste water reuse in ready-mixed concrete production enterprises", focusing on the major elements including Al, Ca, Fe, Mg, S, and Si.

### 2.3.3. *Cement mortar test program*

In this study, to investigate the effect of carbonated concrete wash water (CCWW) on the performance of cement mortar, the test program was designed according to the JTG 3420-2020 "Testing Methods of Cement and Concrete for Highway Engineering". The performance tests for cement mortar included the flow test, the dry shrinkage test, and the strength test. A reference group using potable water (REF) was established to study the effects of uncarbonated, carbonated, and partially carbonated concrete wash water on cement mortar. The test program included uncarbonated concrete wash water (UN), fully carbonated concrete wash water (TH), and partially carbonated concrete wash water (TL). After demolding, the mortar specimens were cured under standard curing conditions at a temperature of  $20 \pm 1$  °C and a relative humidity of  $90 \pm 1\%$  until the specified testing ages.

For clarity, the sample nomenclature used in this study is defined as follows: REF denotes the reference mortar prepared with potable water; UN denotes mortar prepared

with uncarbonated concrete wash water; TL and TH denote mortar prepared with partially carbonated and fully carbonated concrete wash water, respectively; TLR and THR denote the corresponding partially carbonated and fully carbonated wash-water groups with cement reduction, in which the cement content was reduced by an amount equivalent to the solids introduced by the wash water. In addition, THR-0%, THR-25%, THR-50%, THR-75%, and THR-100% represent the groups with different replacement ratios of potable water by fully carbonated concrete wash water under the cement-reduction condition. These groups were designed to distinguish the effects of carbonation degree, the potential contribution of carbonation-derived solids to cement replacement, and the feasible replacement ratio of potable water by CCWW in the mortar system.

To study the effect of partially and fully carbonated concrete wash water on the strength properties of cement mortar, the test program included partially carbonated concrete wash water (TL), with the parameters for partially and fully carbonated concrete wash water given in Table 2. In addition, to investigate whether the carbonation of concrete wash water solids could replace cement and reduce the amount of cement used, further test programs were set up. These included partially carbonated concrete wash water with cement reduction (TLR) and fully carbonated concrete wash water with cement reduction (THR). The test ratios for cement mortar are shown in Table 3.

Finally, to determine the optimal amount of concrete wash water substitution, different levels of substitution of fully carbonated concrete wash water for potable water

with partial cement reduction were tested. The test ratios for cement mortar with different levels of substitution (THR-0%/25%/50%/75%/100%) are shown in Table 4.

**Table 2** Treatment results of CWW with different carbonation parameters

| Condition                      | Partially carbonated | Fully carbonated |
|--------------------------------|----------------------|------------------|
| Hydration duration (h)         | 3                    | 3                |
| Pump flowrate (LPM)            | 48.4                 | 48.4             |
| Treatment duration (min)       | 30                   | 60               |
| CO <sub>2</sub> flowrate (LPM) | 2.5                  | 5                |

**Table 3** Mix ratio of cement mortar

| Sample | Cement (g) | Water (g) | Concrete wash water (g) | Sand (g) |
|--------|------------|-----------|-------------------------|----------|
| REF    | 450        | 225       | 0                       | 1350     |
| UN     | 450        | 0         | 258.75                  | 1350     |
| TL     | 450        | 0         | 258.75                  | 1350     |
| TH     | 450        | 0         | 258.75                  | 1350     |
| TLR    | 416.25     | 0         | 258.75                  | 1350     |
| THR    | 416.25     | 0         | 258.75                  | 1350     |

**Table 4** Mix ratio of cement mortar with different levels of substitution

| Sample   | Cement (g) | Water (g) | Concrete wash water (g) | Sand (g) |
|----------|------------|-----------|-------------------------|----------|
| THR-0%   | 450        | 225       | 0                       | 1350     |
| THR-25%  | 441.57     | 168.75    | 64.68                   | 1350     |
| THR-50%  | 433.13     | 112.50    | 129.37                  | 1350     |
| THR-75%  | 427.40     | 56.25     | 194.05                  | 1350     |
| THR-100% | 416.25     | 0         | 258.75                  | 1350     |

### 3. Carbonation effect analysis of concrete wash water

#### 3.1. Concrete wash water carbonation process characterization

Fig. 4 shows the variation curve of pH value, temperature and mass in the carbonation process of the fully carbonated concrete wash water (TH) (solids content 150:1000, no cooling) of concrete wash water (CWW).

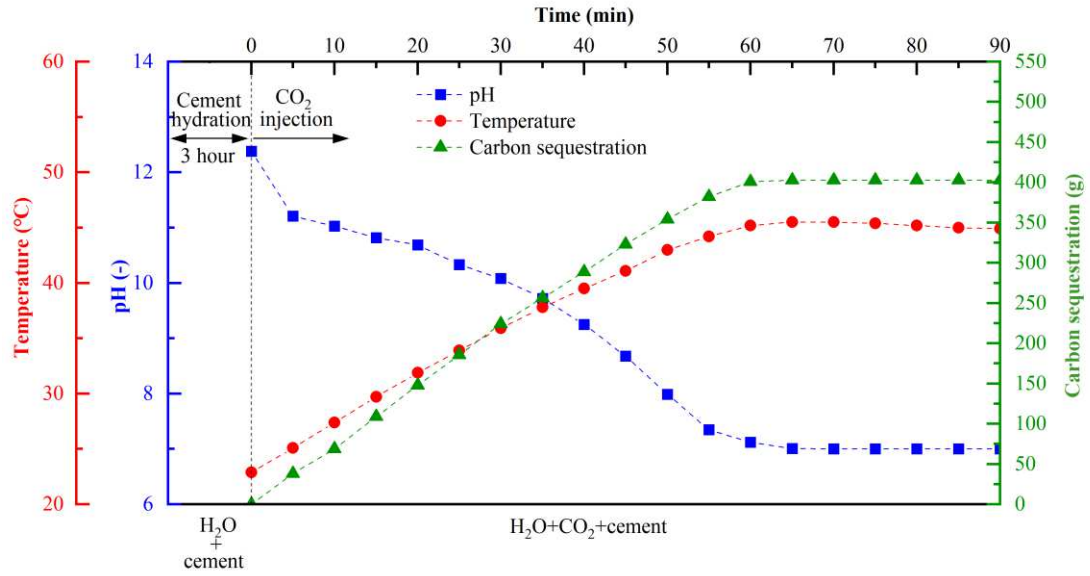


**Fig. 4** The variation curve of pH value, temperature and mass in the carbonation process

The profile shows that the pH dropped steeply from 12.38 to 6.49 within the first 60 min of accelerated carbonation and thereafter plateaued. It should be noted that this low terminal value reflects the high CO<sub>2</sub> uptake capacity of the CWW under investigation; for field concrete, however, carbonation would be arrested at pH  $\approx$  8 to preserve the passivation layer and long-term durability. Moreover, the carbonation reaction is an exothermic reaction, during which the temperature can increase by more than 20 °C; when the pH value reaches a stable value, the temperature rises to the highest point and then begins to decrease. At this point, the active ingredients in the CWW are basically consumed, and the carbonation reaction is complete. At this point, the quality of the CWW after carbonation increases by 406.1g (The CO<sub>2</sub> uptake was quantified gravimetrically with an electronic scale.).

### 3.2. Effect of carbonation on solid-liquid phase of concrete wash water

When CO<sub>2</sub> is introduced into the CWW, it reacts with hydration products, resulting in significant changes in the physical and chemical properties of the CWW. These

changes directly indicate the carbon fixation capacity of the CWW and its effect on the performance of the cement mortar. This study first examines the changes in composition, particle size, and carbon fixation capacity before and after the reaction through solid phase analysis. Additionally, the reaction between  $\text{CO}_2$  and  $\text{Ca}^{2+}$  ions in the CWW leads to the formation of  $\text{CaCO}_3$  precipitates, which significantly changes the ionic concentrations in the CWW. These ionic changes are analyzed by liquid phase analysis.

### *3.2.1. Solid phase analysis*

The samples of solid phase analysis were X-ray diffraction (XRD) spectra of raw cement (OPC), CWW before carbonation (UN) and CWW after carbonation (TH) solid samples.

From Fig. 5, it can be seen that the cement before hydration contains calcium silicate ( $\text{C}_3\text{S}$ ),  $\text{CaO}$  and other substances. After hydration,  $\text{C}_3\text{S}$  and other compounds participate in the hydration reaction to form  $\text{Ca}(\text{OH})_2$ . After carbonation,  $\text{C}_3\text{S}$  is almost completely converted and  $\text{Ca}(\text{OH})_2$  is completely consumed, the diffraction peak of  $\text{CaCO}_3$  is obviously enhanced and a large amount of  $\text{CaCO}_3$  is produced in the reaction. Quantitative analysis shows that  $\text{CaCO}_3$  increases from 5.2% to 23.0%, an increase by 17.8%, and  $\text{Ca}(\text{OH})_2$  decreases from 5.1% to 0%. After carbonation, the CWW stabilizes and other hydration products, forming a stable CCWW.

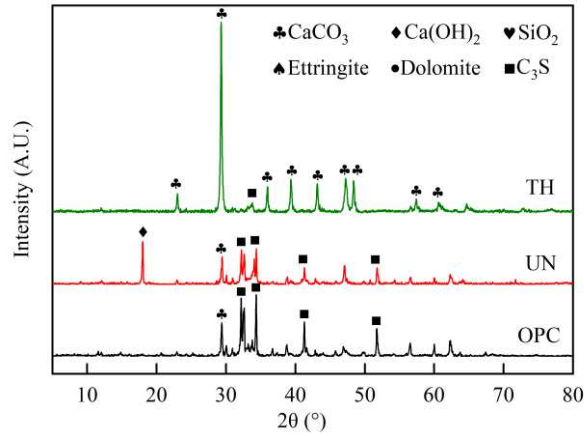


Fig. 5 XRD spectra of solid samples

The corresponding TG-DTG curve is shown in Fig. 6. It can be seen that the original cement powder, when calcined at high temperatures, shows almost no loss of mass in the TG curve.

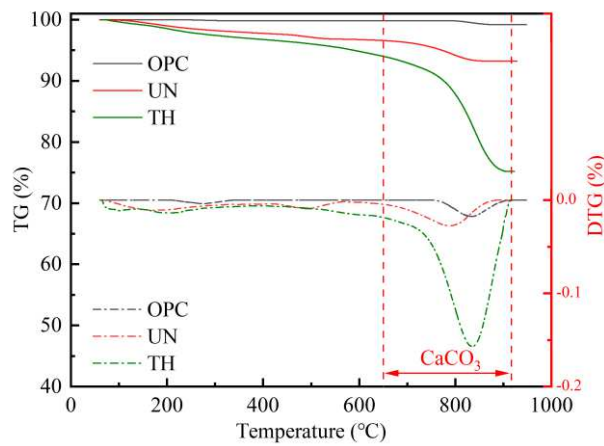


Fig. 6 TG-DTG curve of solid sample

After hydration, there are three curve peaks in the DTG curve. The first peak, about 200°C, is mainly attributed to ettringite. The second peak, around 500°C, which is attributed to the dehydroxylation of  $\text{Ca}(\text{OH})_2$ . The third peak is very obvious, represents the mass loss due to thermal decomposition of  $\text{CaCO}_3$  at this temperature. The data indicate that after hydration, the composition of the CWW reacts with  $\text{CO}_2$  in the air to produce a small amount of  $\text{CaCO}_3$ . After carbonation, a large amount of  $\text{CaCO}_3$  is

produced, so the  $\text{CaCO}_3$  peak is significantly enhanced. Thermogravimetric analysis shows that the content of  $\text{CaCO}_3$  in the uncarbonated concrete wash water (UCWW) sample was 3.49%, whereas the content of  $\text{CaCO}_3$  in the CCWW sample was 19.60%, an increase of 16.11%. These results are in close agreement with the quantitative XRD analysis discussed above.

Fig. 7 shows the particle size analysis curves for three solid powders, showing the particle size distribution and the proportion of particles with different diameters (D) by volume content. The analyses were performed with a BT-9300LD laser diffraction particle-size analyzer.

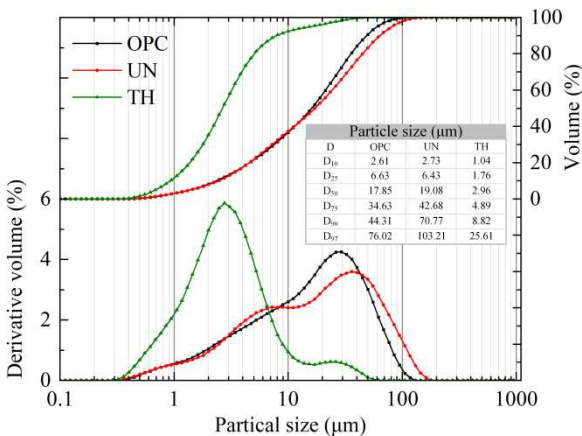


Fig. 7 Particle size curve and particle content of different particle sizes

The figure shows that the particle size of OPC particles increases slightly after hydration, indicating that the particle size of the product after hydration is larger than that of OPC particles. After carbonation, the particle size is significantly reduced, with particles larger than 100  $\mu\text{m}$  disappearing completely. The particle size of the CCWW is reduced to less than 40  $\mu\text{m}$ , which is significantly smaller than the maximum particle size of OPC. Specifically, after carbonation, the  $D_{50}$  (median particle size, indicating that 50% of the particles are smaller than this value) of the CWW decreased from 19.08

$\mu\text{m}$  to  $2.96 \mu\text{m}$ , while the  $D_{97}$  decreased from  $103.21 \mu\text{m}$  to  $25.61 \mu\text{m}$ . This indicates that carbonation refines the solid particles in the CWW, concentrating them to sizes below  $26.0 \mu\text{m}$ . This refinement of the particles has a good filling effect(Jiang et al., 2023), which can be further verified by pore tests.

Fig. 8 shows SEM images of the solid phase before and after carbonation at 5000x and 15000x magnification respectively. Composition was determined by EDS analysis, with the major components labelled in the images.

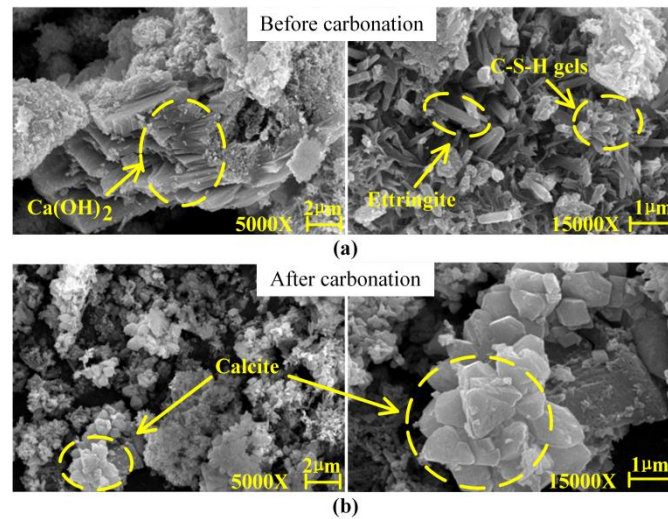


**Fig. 8** SEM images of solid sample (a) Before carbonation 5000 $\times$  and 15000 $\times$  (b) After carbonation 5000 $\times$  and 15000 $\times$

Fig. 8(a) shows the solid sample from the CWW before carbonation, where the composition is mainly cement hydration products. The needle-like structures correspond to ettringite, the hexagonal plate-like layered structures are  $\text{Ca}(\text{OH})_2$  and the cloudy and reticular material represents C-S-H gel(Hu and Li, 2014). After carbonation, as shown in Fig. 8(b), the needle-like ettringite, hexagonal plate-like calcium hydroxide and cloud-like C-S-H gel are no longer present. Instead, a significant amount of fine-grained calcium carbonate (calcite) is precipitated in the CCWW. These mineralogical results, together with the significant decrease in  $\text{Ca}^{2+}$  concentration in the

liquid phase after carbonation, support that the major reactive alkaline components had been substantially consumed when the pH approached a stable neutral range.

### 3.2.2. Liquid analysis

The liquid phase analysis samples include CWW before carbonation and the CWW sample after carbonation. As shown in Table 5, the concentration of  $\text{Ca}^{2+}$  in the CWW decreases significantly after carbonation, mainly due to the formation of stable  $\text{CaCO}_3$ . A small residual amount of  $\text{Ca}^{2+}$  may result from the reaction of excess  $\text{CO}_2$  with  $\text{CaCO}_3$  in solution to form  $\text{Ca}(\text{HCO}_3)_2$ . The Fe content of the CWW remains largely unchanged after carbonation, while the concentrations of Al, Mg, S and Si increase. This observation is consistent with the XRD analysis of the solid phase changes before and after carbonation, which indicates that  $\text{Ca}(\text{OH})_2$  and  $\text{C}_3\text{S}$  are almost completely consumed in the solid phase. Carbonisation and decomposition of ettringite contribute to an increased concentration of Al in the solution. In addition, the decomposition of dolomite and the presence of free MgO in an acidic environment leads to an increased Mg content. The carbonation of AFt and  $\text{C}_3\text{S}$  also leads to higher concentrations of S and Si in the solution. Table 6 shows the test results for CWW quality before and after carbonation, showing that all indicators comply with the DB51/T 2681-2020 "Technical Regulations on Recycling of Wastewater and Waste Slurry from Ready-Mix Concrete Batching Plant". The slight decrease in the  $\text{Cl}^-$  concentration, coupled with the increase in the  $\text{SO}_4^{2-}$  concentration, suggests that several effects are at work.  $\text{CO}_2$  may degrade the original sulphate-containing hydration products, and the decrease in pH may further disrupt the sulphate-containing phases. In

addition, the formation of calcium carbonate from  $\text{Ca}^{2+}$  reduces the availability of calcium required for gypsum formation.

**Table 5** Comparison of ion concentration in CWW before and after carbonation

| Element   | Before Carbonation | After Carbonation |
|-----------|--------------------|-------------------|
| Al (mg/L) | 0.02               | 0.31              |
| Ca (mg/L) | 1334.4             | 41.1              |
| Fe (mg/L) | 0.1                | 0.002             |
| Mg (mg/L) | 3.3                | 455.9             |
| S (mg/L)  | 371.7              | 652.2             |
| Si (mg/L) | 0.5                | 84.5              |

**Table 6** Quality requirements of CWW

| Item                      | Control Indicator | Before Carbonation | After Carbonation |
|---------------------------|-------------------|--------------------|-------------------|
| pH                        | $\geq 4.5$        | 12.89              | 6.51              |
| Insoluble matter (mg/L)   | $\leq 2000$       | -                  | -                 |
| Soluble matter (mg/L)     | $\leq 5000$       | 294.23             | 69.52             |
| $\text{Cl}^-$ (mg/L)      | $\leq 1000$       | 33.25              | 50.17             |
| $\text{SO}_4^{2-}$ (mg/L) | $\leq 2000$       | 940.10             | 1452.36           |
| Alkali content (mg/L)     | $\leq 1500$       | 479.44             | 435.43            |

### 3.3. Performance test of cement mortar

In order to evaluate the effect of CWW on cement mortar, the working performance of cement mortar was tested. Among them, REF is the control group using potable water, UN is the use of uncarbonated concrete wash water, and TH is the use of completely carbonated concrete wash water.

#### 3.3.1. Setting time

It can be seen from Fig. 9 that the initial setting and final setting of cement mortar mixed with UCWW are accelerated, whereas those of cement mortar mixed with CCWW are prolonged. The acceleration of cement hydration in the presence of UCWW can be attributed to its solid phase, which contains hydration products such as calcium ions, calcium hydroxide and C-S-H. These components provide additional reactants for cement setting. These components provide additional reactants that promote cement

setting(Chen et al., 2018). After carbonation, the solid phase consists mainly of inert calcium carbonate particles which reduce the effective cement content. This reduction reduces the hydration water requirement, increases the water/cement ratio and delays cement hydration.

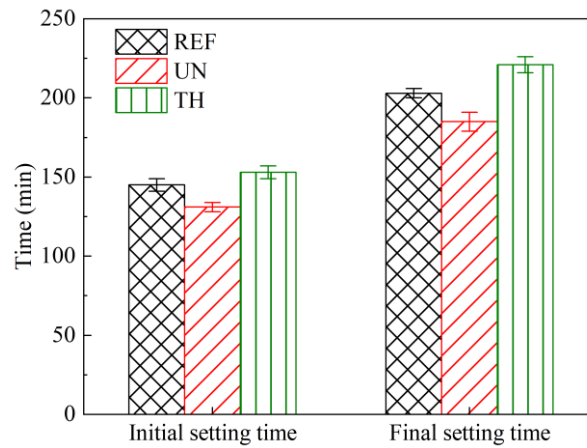


Fig. 9 Setting time of cement mortar

### 3.3.2. Flowability

The flowability of cement is a critical factor influencing the performance of concrete during construction and after curing. As shown in Fig. 10, the flowability of the reference mortar was 198 mm, while that of the UCWW and CCWW groups decreased to 176 mm and 173 mm, respectively. Compared with the reference group, the flowability of the CCWW group decreased by 12.6%. Both the UCWW and CCWW groups fell below the limit of 180 mm. This reduction is attributed to the production of fine calcium carbonate particles in the CCWW, which increases the specific surface area and water demand, thereby reducing flowability(Feng et al., 2022).

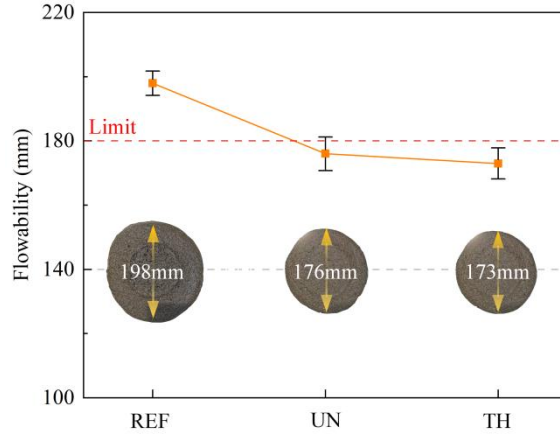


**Fig. 10** Flowability of cement mortar

To further support this interpretation, the particle size distribution results shown in Fig. 7 indicate that carbonation significantly refined the solid particles in CWW. After carbonation, the  $D_{50}$  decreased from  $19.08\mu\text{m}$  to  $2.96\mu\text{m}$ , while the  $D_{97}$  decreased from  $103.21\mu\text{m}$  to  $25.61\mu\text{m}$ . The formation of finer  $\text{CaCO}_3$ -rich particles increased the specific surface area and water demand of the mortar system, which further contributed to the lower flowability of CCWW.

### 3.3.3. Drying shrinkage

Studies indicate that the main factors influencing the drying shrinkage of portland cement mortar are the cement content and the water-cement ratio (Ye et al., 2017). Higher portland cement content typically results in higher drying shrinkage of cement specimens.

As shown in Fig. 11, the drying shrinkage rate of the cement mortar specimens in the potable-water reference group was 0.140% after 28 days. In contrast, specimens using UCWW showed a drying shrinkage rate of 0.158%, while those using CCWW showed a drying shrinkage rate of 0.066%, corresponding to a 52.9% reduction relative to the potable-water reference group. According to the standard GB/T 17671-1999

"Standard for Test Methods of Cement Mortar Strength (ISO Method)", the shrinkage rate should be less than 0.15%; the specimen using UCWW does not meet the requirement, while the specimen using CCWW meets the requirement. These results demonstrate that replacing mixing water with CCWW can improve the drying shrinkage performance of cement mortars.

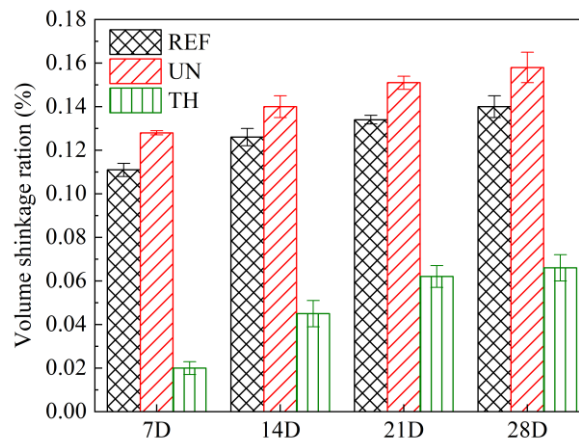


Fig. 11 Dry shrinkage of cement mortar

#### 3.3.4. Mechanical property

Fig. 12(a) shows that the addition of UCWW reduces the compressive strength of cement mortar. However, when the CWW is treated with CO<sub>2</sub>, the compressive strength gradually increases with longer treatment times, indicating that carbonation mitigates the adverse effects of CWW on compressive strength. Compared to the control group, the compressive strength increased by 3.3% at 3 days, 11.8% at 7 days and 13.0% at 28 days. In the two reduced cement groups, the compressive strength decreased significantly and fell below that of the control group. This suggests that the level of cement reduction in this test, set at 7.5%, was excessive. In future tests, consideration should be given to partially replacing the mixing water with CWW to ensure that the compressive strength meets standard requirements whilst reducing the cement content.

As shown in Fig. 12(b), the flexural strength of the cement mortar samples was lower than that of the control group at all ages after the addition of CWW, regardless of whether carbon treatment was applied. This suggests that the use of CWW had a negative effect on the flexural strength of cement mortar. Porosity is a key factor affecting the mechanical properties of cementitious materials and can be analyzed through the pore-size distribution. As shown in Fig. 13, both the 3-day and 28-day tests followed the same trend, with the pore distribution ranked as UN > REF > TH. The lower flexural strength of the UN group may therefore be associated with its relatively larger internal pore structure. For the TH group, although carbonation generated fine  $\text{CaCO}_3$  particles that could fill part of the pores and improve matrix compactness, the influence of the water-to-binder condition on flexural behavior appeared to be more dominant, so the flexural strength still remained lower than that of the control group.

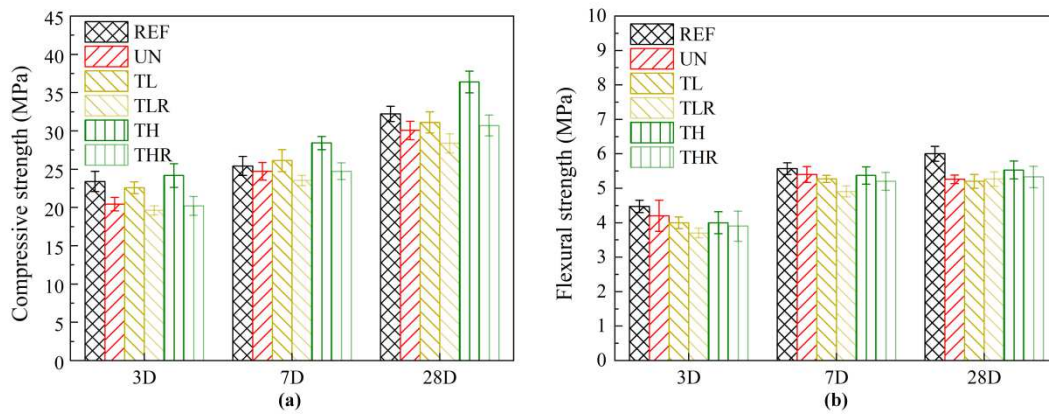


**Fig. 12** Effect of CCWW on the strength properties of cement mortar

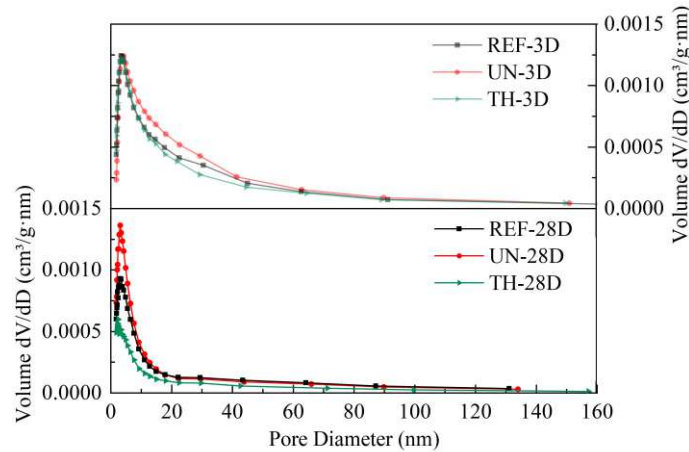


**Fig. 13** Pore size distribution of cement mortar

This indicates that the use of CCWW significantly refines the pores compared to the control, and the use of UCWW increases the pores. The more significant pore reduction in 28D compared to 3D was due to more complete hydration reaction with increasing age and more hydration products, which filled the pores. In addition, carbonation altered the solid-phase composition of CWW and generated a large amount of fine  $\text{CaCO}_3$  particles. As shown in Fig. 8, these carbonation-derived particles were relatively fine and uniformly distributed, while the pore size distribution results in Fig. 13 indicate that the CCWW group exhibited a lower pore volume than the REF and UCWW groups. These observations suggest that the improved compressive strength is closely associated with pore refinement and matrix densification. The filling effect of fine  $\text{CaCO}_3$  particles is considered to make an important contribution to this microstructural improvement.

In addition to our findings, the study by CarbonCure Technologies have demonstrated that  $\text{CO}_2$  upcycling can be effectively integrated into concrete production at an industrial scale. During their trials,  $\text{CO}_2$  injected into concrete mixers resulted in an average uptake efficiency of 88%, which contributed to a reduction in the carbon

footprint of the blocks by 1.4%. Furthermore, the process led to significant improvements in compressive strength (13-33% increase) and reductions in water absorption (18-36%)(Monkman and MacDonald, 2016). This work underscores the potential for CO<sub>2</sub> sequestration within industrial-scale concrete production systems.

#### *3.4. Concrete wash water substitution test*

As the above CWW completely replaces potable water and reduces the compressive strength of the experimental cement group, it is not suitable for engineering applications. In addition, its flowability does not meet the required standards. To address these issues, a test group with varying levels of CCWW substitution was designed to determine the optimum level of substitution that would meet the minimum standard requirements.

As shown in Fig. 14, the test results for cement mortar performance at different levels of CCWW substitution are presented. In Fig. 14(a), flowability below the minimum requirement of 180 mm was observed in the 75% and 100% CCWW substitution test groups. Fig. 14(b) shows that the drying shrinkage rate is improved in the test groups containing CCWW. In the compressive strength test shown in Fig. 14(c), the 28-day strength of the 75% and 100% substitution test groups did not meet the minimum requirement of 42.5 MPa, whereas Fig. 14(d) shows that the flexural strength of all test groups met the standard. Therefore, to ensure that the use of CCWW meets the actual project requirements, it is crucial to control the degree of substitution. In this study, a substitution level of 50% was found to meet the requirements.

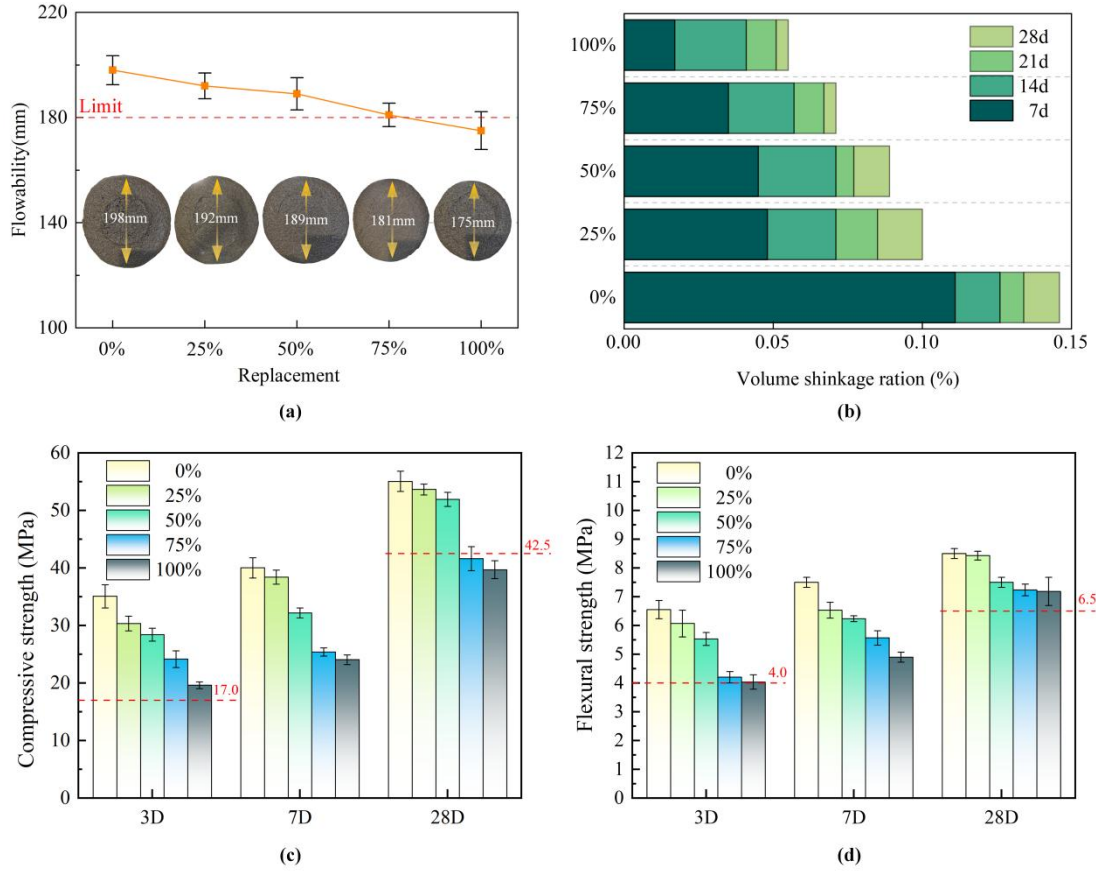


Fig. 14 Properties of cement mortar at different substitution rates

#### 4. Study on the maximum CO<sub>2</sub> absorption rate of carbon sequestration device based on RSM

Response Surface Methodology (RSM) is a powerful statistical technique used to optimise process conditions and develop models (Kaliyavaradhan et al., 2020). It allows the evaluation of the effects of different experimental factors and their interactions on one or more response variables. The relationship between the experimental variables and the response variables can be modelled by regression of a second order polynomial as described in Eq. (5).

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \beta_{ij} X_i X_j + \varepsilon \quad (5)$$

where,  $Y$  = predictive response,  $\beta_0$  = intercept,  $\beta_i$ ,  $\beta_j$  = linear effect coefficient,  $\beta_{ii}$ ,  $\beta_{ij}$  =

quadratic effect coefficient,  $\beta_{ij}$ = interaction effect coefficient,  $X_i$ ,  $X_j$ = independent variable,  $\varepsilon$ = residual. This section is intended to optimize the operating conditions of the carbonation process and identify the key factors affecting CO<sub>2</sub> absorption rate, so as to provide a process basis for the efficient preparation of CCWW.

Three main factors, solid-liquid ratio, CO<sub>2</sub> gas flow rate, and coolant temperature, were identified from the previous single-factor test and three levels were determined. In this study, Box-Behnken Design (BBD) optimisation was used to evaluate the CO<sub>2</sub> absorption rate of CWW. The solid/liquid ratio is represented by the coded value  $X_1$ , with corresponding low and high values of 100:1000 and 200:1000, respectively. The CO<sub>2</sub> gas flow rate is represented by  $X_2$  with corresponding low and high values of 3 LPM and 11 LPM. The coolant temperature is represented by  $X_3$ , with corresponding minimum and maximum values of 0°C and 30°C, as shown in Table 7.

**Table 7** Factors and factor levels adopted for RSM

| Factor                                 | Code  | Factor levels of code |          |          |
|----------------------------------------|-------|-----------------------|----------|----------|
|                                        |       | -1                    | 0        | 1        |
| Solid/Liquid ratio<br>(-)              | $X_1$ | 100:1000              | 150:1000 | 200:1000 |
| CO <sub>2</sub> gas flow rate<br>(LPM) | $X_2$ | 3                     | 7        | 11       |
| Coolant<br>temperature (°C)            | $X_3$ | 0                     | 15       | 30       |

#### 4.1. Response surface analysis

In this study, three factors and three levels were selected for BBD experiments, resulting in a total of 17 experimental runs Table 8. In each experiment, the amount of CO<sub>2</sub> carbonated was measured using an electronic scale and the time taken for the pH of the CWW to stabilise below 7 was recorded. The effects of solid/liquid ratio, gas flow rate and temperature on the CO<sub>2</sub> absorption rate of the CWW were evaluated using

RSM, and a statistical model was developed to predict the maximum CO<sub>2</sub> absorption rate. The CO<sub>2</sub> absorption rate was calculated as follows:

$$AR_{CO_2} = \frac{m_{CO_2}}{T} \quad (6)$$

where,  $AR_{CO_2}$  is the CO<sub>2</sub> absorption rate;  $m_{CO_2}$  is the mass of carbon dioxide uptake after carbonation;  $T$  is the total time from the start of the carbonation reaction with carbon dioxide injection until the pH drops below 7 and stabilizes.

**Table 8** Factor combinations and response

| run | $X_1$ : Solid/Liquid<br>ratio (-) | $X_2$ : CO <sub>2</sub> gas flow<br>rate (LPM) | $X_3$ : Coolant<br>temperature (°C) | $AR_{CO_2}$ (g/min) |
|-----|-----------------------------------|------------------------------------------------|-------------------------------------|---------------------|
| 1   | 150:1000                          | 7                                              | 15                                  | 10.1                |
| 2   | 100:1000                          | 7                                              | 30                                  | 7.6                 |
| 3   | 150:1000                          | 3                                              | 30                                  | 5.2                 |
| 4   | 150:1000                          | 11                                             | 0                                   | 16.3                |
| 5   | 150:1000                          | 11                                             | 30                                  | 15.2                |
| 6   | 150:1000                          | 7                                              | 15                                  | 10.2                |
| 7   | 200:1000                          | 3                                              | 15                                  | 5.3                 |
| 8   | 150:1000                          | 7                                              | 15                                  | 10.1                |
| 9   | 100:1000                          | 7                                              | 0                                   | 8.6                 |
| 10  | 150:1000                          | 3                                              | 0                                   | 6.3                 |
| 11  | 200:1000                          | 7                                              | 0                                   | 10.2                |
| 12  | 150:1000                          | 7                                              | 15                                  | 9.5                 |
| 13  | 100:1000                          | 11                                             | 15                                  | 13.6                |
| 14  | 200:1000                          | 7                                              | 30                                  | 8.1                 |
| 15  | 150:1000                          | 7                                              | 15                                  | 10.1                |
| 16  | 200:1000                          | 11                                             | 15                                  | 16.2                |
| 17  | 100:1000                          | 3                                              | 15                                  | 4.0                 |

Table 9 shows the analysis of variance (ANOVA) of CO<sub>2</sub> uptake (%) from the RSM analysis. Where variables A, B, and C denote the solid/liquid ratio, CO<sub>2</sub> gas flow rate, and coolant temperature, respectively. The interactions of A with B, A with C, and B with C are denoted as AB, AC, and BC, respectively. The secondary effects of A, B, and C are denoted by A<sup>2</sup>, B<sup>2</sup>, and C<sup>2</sup>, respectively. The P<0.0001 of the model can be considered significant for this model, thus the statistical model proposed in this study

is significant, the lack-of-fit term  $P=0.0773>0.05$  can be considered as non-significant for the lack-of-fit term, which indicates that the model is well simulated and the model correlates well with the experimental data without the need to make adjustments (Senthil Kumar and Baskar, 2014, Tie et al., 2022). The adjusted R-squared value ( $\text{Adj } R^2 = 0.9862$ ) indicates that the model explains 98.62% of the variation in the response variable. The predicted R-squared ( $\text{Pred } R^2 = 0.9218$ ), which is close to the adjusted R-squared, further supports the accuracy of the model. The signal to noise ratio,  $\text{Adeq Precision} = 35.1441 (>4)$ , indicates that the reliability and accuracy of the test is satisfactory. In Table 8, factors A, B, C, AB,  $A^2$  and  $B^2$  are significant model terms, while the remaining terms are considered insignificant.

**Table 9** ANOVA for  $\text{CO}_2$  uptake rate

| Source      | Sum of Squares | df | Mean Square | F-value | p-value  | Remark          |
|-------------|----------------|----|-------------|---------|----------|-----------------|
| Model       | 249.87         | 9  | 27.76       | 128.07  | < 0.0001 | Standard        |
| A           | 6.3            | 1  | 6.3         | 29.07   | 0.001    | Deviation =     |
| B           | 226.84         | 1  | 226.84      | 1046.4  | < 0.0001 | 0.4656          |
| C           | 3.51           | 1  | 3.51        | 16.2    | 0.005    | Mean = 9.92     |
| AB          | 1.44           | 1  | 1.44        | 6.64    | 0.0366   | Coefficient of  |
| AC          | 0.3025         | 1  | 0.3025      | 1.4     | 0.2761   | Variation % =   |
| $A^2$       | 5.69           | 1  | 5.69        | 26.25   | 0.0014   | 4.69            |
| $B^2$       | 6.19           | 1  | 6.19        | 28.55   | 0.0011   | $R^2 = 0.9940$  |
| $C^2$       | 0.1901         | 1  | 0.1901      | 0.877   | 0.3802   | Adjusted $R^2$  |
| Residual    | 1.52           | 7  | 0.2168      |         |          | = 0.9862        |
| Lack of Fit | 1.2            | 3  | 0.3992      | 4.99    | 0.0773   | Predicted $R^2$ |
| Pure Error  | 0.32           | 4  | 0.08        |         |          | = 0.9218        |
| Cor Total   | 251.39         | 16 |             |         |          | Adequate        |
|             |                |    |             |         |          | Precision       |
|             |                |    |             |         |          | = 35.1441       |

To check the reliability of the model, the carbon sequestration rate was fitted and

the correlation coefficient between the measured and predicted values was 0.9940, indicating a good fit. When the  $X_3$  value in the prediction model is set to 0, the effect of the interaction between the solid/liquid ratio and the  $\text{CO}_2$  gas flow rate on the  $\text{CO}_2$  absorption rate can be observed. As shown in Fig. 15, as the solid/liquid ratio increases from 100:1000 to 200:1000, the carbon sequestration rate initially increases and then decreases. Conversely, as the  $\text{CO}_2$  gas flow rate increases from 3 to 11 LPM, the carbon sequestration rate increases steadily. This behaviour can be attributed to the increase in cement content in the scrubbing water at higher solid/liquid ratios, which provides more  $\text{Ca}^{2+}$  ions to react with  $\text{CO}_2$ , thereby increasing the reaction rate. However, the reaction generates significant heat, which raises the temperature of the CWW. Studies have shown that in the 40-80°C range, the reduction in  $\text{CO}_2$  solubility has a greater effect on carbonation, slowing down  $\text{CO}_2$  dissolution and thereby reducing the reaction rate. As the  $\text{CO}_2$  gas flow rate increases, more dissolved  $\text{CO}_2$  is available to participate in the reaction, resulting in an increased reaction rate.

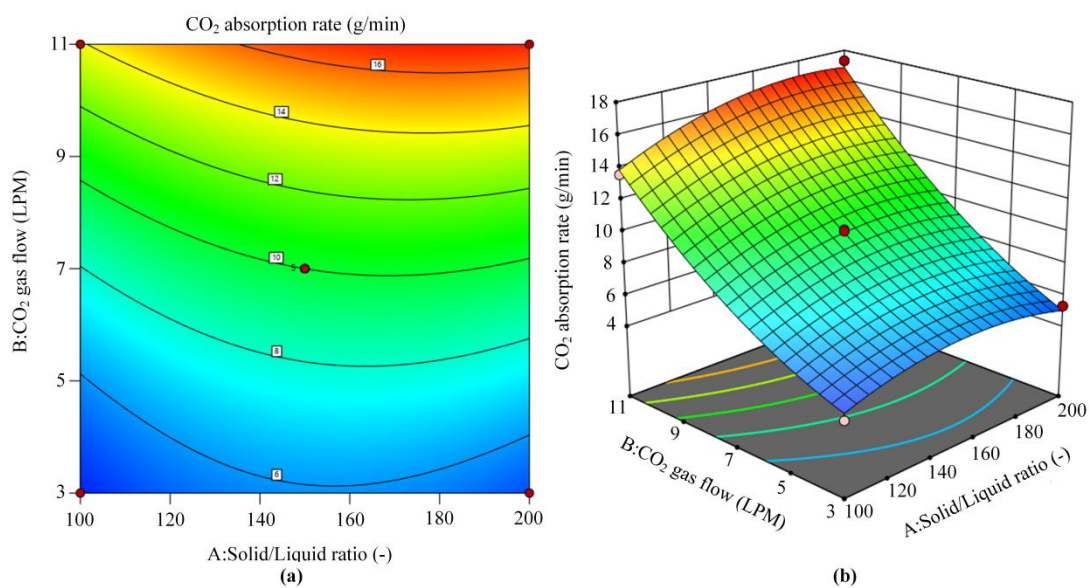


**Fig. 15** (a) 2-D contour plot of response and (b) 3-D response surface plot.

In summary, the RSM model effectively predicts the CO<sub>2</sub> absorption rate of concrete wash water (CWW) within the range of the research factors. The established model can accurately predict the CO<sub>2</sub> absorption rate based on the actual factors as shown in Eq. (7).

$$Y = 10 + 0.8875X_1 + 5.3300X_2 - 0.6625X_3 + 0.6000X_1X_2 - 0.2750X_1X_3 - 1.1600X_1^2 + 1.2100X_2^2 - 0.2125X_3^2 \quad (7)$$

where, X<sub>1</sub> is solid/liquid ratio, X<sub>2</sub> is CO<sub>2</sub> gas flow rate, X<sub>3</sub> is coolant temperature.

#### 4.2. Test data verification

The BBD optimisation analysis of the carbon sequestration operating parameters was performed and the optimal combination of parameters to maximise the CO<sub>2</sub> absorption rate was identified (solid/liquid ratio of 178:1000, CO<sub>2</sub> gas flow rate of 11 LPM and coolant temperature of 2.7°C). Under these optimised conditions, the average carbon sequestration rate was 18.6 g/min, an increase of 14.1% over the maximum carbon sequestration rate observed in the previous test.

To further validate the model's predictive accuracy, eleven additional operational-parameter combinations were randomly selected for carbon-sequestration experiments, with each combination being run in triplicate. The predicted values from the model were compared with the actual measured values. The results are shown in Fig. 16. Although the predicted values are not exactly identical to the measured values, they are generally close, with errors consistently within 10%. This confirms that the model has a good prediction accuracy.

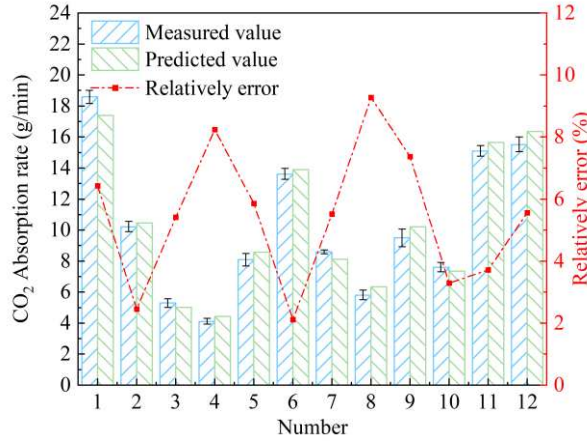


**Fig. 16** Comparison between predicted and measured CO<sub>2</sub> absorption rates for model validation.

## 5. Conclusions

In pursuit of diminishing the carbon footprint associated with cement-based materials, this study sought to ascertain the viability of recycling concrete wash water (CWW). The investigation was designed to assess the efficacy of a highly efficient carbon capture methodology for the carbonation treatment of CWW, with the aim of facilitating its effective reuse. Through microanalysis, the study identified alterations in the composition of the CWW, and a series of cement mortar tests were devised to evaluate the impact of carbonated concrete wash water (CCWW) on the performance characteristics of cement mortar. Ultimately, Response Surface Methodology (RSM) was utilized to refine the operational parameters of the carbonation apparatus, thereby enhancing the absorption rate of carbon dioxide within the CWW. The principal conclusions of the study are as follows:

(1) The analysis of the CWW composition indicates that after carbonation, the hydration components within the cement react with carbon dioxide to form a substantial amount of small and stable calcite-type calcium carbonate particles. These particles fill the micropores of the cement mortar, thereby enhancing its compressive strength.

(2) Compared with the potable-water reference group, cement mortars prepared with CCWW showed a modified setting behavior, characterized by an accelerated initial setting time and a delayed final setting time. In addition, drying shrinkage was reduced by 52.9%, and compressive strength was increased by 13.0%, which was associated with pore refinement and matrix densification. However, the flowability decreased due to the increased water demand caused by the fine calcium carbonate particles.

(3) The experimental results utilizing varying proportions of CCWW as a replacement for potable water in cement mortar mixtures demonstrate that when the replacement ratio of CCWW is less than 50% of the total water content in the cement mortar mixture, the flowability, shrinkage, compressive strength, and flexural strength of the cement mortar meet the performance requirements for practical applications.

(4) Three key operating parameters affecting the CO<sub>2</sub> absorption rate of the CWW were optimized using RSM. The results indicated that the optimized CO<sub>2</sub> absorption rate increased by 14.1%, reaching 18.6 g/min, under the conditions of a solid/liquid ratio of 178:1000, a gas flow rate of 11 LPM, and a coolant temperature of 2.7°C.

## **6. Future work**

It should be noted that the present study mainly focused on the carbonation mechanism of concrete wash water and the preliminary evaluation of its reuse performance in the cement mortar system, rather than a complete engineering-performance validation. Due to the limitations of the current experimental conditions, durability-related properties were not systematically investigated in this work. This

study primarily focuses on the cement mortar system and does not involve the exploration of concrete system performance. Given that the presence of coarse and fine aggregates in concrete may affect the mechanism of action and mechanical properties of CCWW, future research could further conduct the preparation and performance testing of concrete specimens, with a particular focus on their permeability, frost resistance, and chloride ion penetration resistance. This would provide a more comprehensive scientific basis for the large-scale application of CCWW in practical engineering. In addition, although the influence of chemical admixtures on workability was not systematically investigated in this study, admixture compatibility and particle size distribution are both considered important factors affecting the rheological behavior of mortar and will be further addressed in future work.

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#### **Credit authorship contribution statement**

Dianjun Yang: Writing - original draft, Methodology, Investigation, Data curation. Jiming Lin (corresponding author): Writing - review & editing, Writing - original draft, Methodology, Investigation, Data curation, Conceptualization. Zhiwen Zeng: Writing - review & editing, Validation. Zhiyu Lin: Writing - review & editing, Validation,

Supervision.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

Data will be made available on request.

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